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Polyacetylene,  $(CH)_x$ , is the simplest possible conjugated organic polymer and is therefore of special fundamental interest. In a series of studies, Shirakawa and Ikeda have shown that it can be prepared in the form of lustrous, silvery, flexible, polycrystalline films having any desired cis/trans content by catalytic polymerization of gaseous acetylene,  $C_2H_2$ . The cis-rich films can be stretched easily at room temperature in excess of three times their original length with concomitant partial alignment of the  $(CH)_x$  fibrils. Dark red gels of toluene in  $(CH)_x$  may be prepared using a lower catalyst concentration. Highly porous, very low density,

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20. "foam-like"  $(CH)_x$  can be obtained from these gels. Both cis- and trans- $(CH)_x$  are p-type semiconductors which can be treated with a variety of p- or n-type dopants with concomitant increase in conductivity to give a series of semiconductors and ultimately, "organic metals." This report will be directed primarily towards description of the more chemically oriented aspects of  $(CH)_x$  and its derivatives.

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Organic Metals and Semiconductors:

The Chemistry of Polyacetylene,  $(CH)_x$ , and its Derivatives

by

Alan G. MacDiarmid and Alan J. Heeger<sup>\*</sup>

Presented at

The 3rd International Congress of Quantum Chemistry  
Kyoto, Japan  
October 1979

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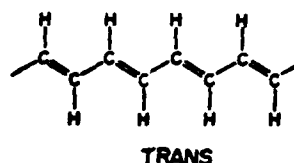
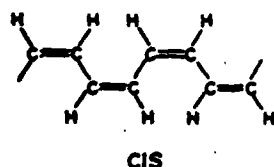
## ORGANIC METALS AND SEMICONDUCTORS:

### THE CHEMISTRY OF POLYACETYLENE, $(CH)_x$ , AND ITS DERIVATIVES

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Polyacetylene,  $(CH)_x$ , is the simplest possible conjugated organic polymer and is therefore of special fundamental interest. In a series of studies, Shirakawa and Ikeda have shown that it can be prepared in the form of lustrous, silvery, flexible, polycrystalline films having any desired cis/trans content by catalytic polymerization of gaseous acetylene,  $C_2H_2$  (1-4):



The cis-rich films can be stretched easily at room temperature in excess of three times their original length with concomitant partial alignment of the  $(CH)_x$  fibrils (5,6). Dark red gels of toluene in  $(CH)_x$  may be prepared using a lower catalyst concentration (7). Highly porous, very low density, "foam-like"  $(CH)_x$  can be obtained from these gels (7). Both cis- and trans- $(CH)_x$  are p-type semiconductors (8) which can be treated with a variety of p- or n-type dopants with concomitant increase in conductivity to give a series of semiconductors and ultimately, "organic metals." This report will be directed primarily towards a description of the more chemically oriented aspects of  $(CH)_x$  and its derivatives.

#### 1. DOPING OF $(CH)_x$ FILMS.

The various types of dopants and doping procedures, the nature of the  $(CH)_x$  chain, and the nature of the dopant in the films will be described below. The terms "cis" and "trans" used in conjunction with a doped film will refer to the principal isomeric composition before doping and does not imply that the isomeric composition either remains constant or changes during the doping process.

##### (A) P-Type Doping.

(1) Dopants and Methods of Doping. When either cis or trans films are exposed to the vapor of electron-attracting substances (p-type dopants) such as

Br<sub>2</sub>, I<sub>2</sub>, AsF<sub>5</sub>, H<sub>2</sub>SO<sub>4</sub>, HClO<sub>4</sub>, etc. (9,10) they become "doped" with the species and their electrical (11) (Table I) and optical (12) properties change markedly.

**TABLE I**  
**DOPANTS FOR (CH)<sub>x</sub>.<sup>a,b</sup>**

	Conductivity (ohm <sup>-1</sup> cm <sup>-1</sup> ) 25°C
<u>cis</u> -(CH) <sub>x</sub>	1.7 x 10 <sup>-9</sup>
<u>trans</u> -(CH) <sub>x</sub>	4.4 x 10 <sup>-5</sup>
<b>A. p-Type (electron-attracting) Dopants</b>	
<u>trans</u> -[CH(HBr) <sub>0.04</sub> ] <sub>x</sub> <sup>c</sup>	7 x 10 <sup>-4</sup>
<u>trans</u> -[CHCl <sub>0.02</sub> ] <sub>x</sub> <sup>c</sup>	1 x 10 <sup>-4</sup>
<u>trans</u> -[CHBr <sub>0.23</sub> ] <sub>x</sub>	4 x 10 <sup>-1</sup>
<u>cis</u> -[CH(ICl) <sub>0.14</sub> ] <sub>x</sub>	5 x 10 <sup>1</sup>
<u>cis</u> -[CHI <sub>0.30</sub> ] <sub>x</sub>	5.5 x 10 <sup>2</sup>
<u>trans</u> -[CHI <sub>0.20</sub> ] <sub>x</sub>	1.6 x 10 <sup>2</sup>
<u>cis</u> -[CH(IBr) <sub>0.15</sub> ] <sub>x</sub>	4.0 x 10 <sup>2</sup>
<u>trans</u> -[CH(AsF <sub>5</sub> ) <sub>0.10</sub> ] <sub>x</sub> <sup>c</sup>	4.0 x 10 <sup>2</sup>
<u>cis</u> -[CH(AsF <sub>5</sub> ) <sub>0.10</sub> ] <sub>x</sub>	1.2 x 10 <sup>3</sup>
<u>cis</u> -[CH <sub>1.1</sub> (AsF <sub>6</sub> ) <sub>0.10</sub> ] <sub>x</sub>	ca. 7 x 10 <sup>2</sup>
<u>cis</u> -[CH(SbF <sub>6</sub> ) <sub>0.05</sub> ] <sub>x</sub>	4.0 x 10 <sup>2</sup>
<u>cis</u> -[CH(SbCl <sub>6</sub> ) <sub>0.009</sub> ] <sub>x</sub>	1 x 10 <sup>-1</sup>
<u>cis</u> -[CH(SbCl <sub>8</sub> ) <sub>0.0095</sub> ] <sub>x</sub>	1 x 10 <sup>1</sup>
<u>cis</u> -[CH(SbCl <sub>5</sub> ) <sub>0.022</sub> ] <sub>x</sub>	2
<u>cis</u> -[CH(BF <sub>2</sub> ) <sub>0.09</sub> ] <sub>x</sub> <sup>d</sup>	1 x 10 <sup>2</sup>
<u>cis</u> -[CH(SO <sub>3</sub> F) <sub>y</sub> ] <sub>x</sub>	7 x 10 <sup>2</sup>
<u>cis</u> -[CH(ClO <sub>4</sub> ) <sub>0.0645</sub> ] <sub>x</sub>	9.7 x 10 <sup>2</sup>
<u>cis</u> -[CH(AsF <sub>4</sub> ) <sub>0.077</sub> ] <sub>x</sub>	2.0 x 10 <sup>2</sup>
<u>cis</u> -[CH <sub>1.011</sub> (AsF <sub>5</sub> OH) <sub>0.011</sub> ] <sub>x</sub> <sup>e</sup>	ca. 7 x 10 <sup>2</sup>
<u>cis</u> -[CH <sub>1.058</sub> (PF <sub>5</sub> OH) <sub>0.058</sub> ] <sub>x</sub> <sup>e</sup>	ca. 3 x 10 <sup>1</sup>
<u>cis</u> -[CH(H <sub>2</sub> SO <sub>4</sub> ) <sub>0.106</sub> (H <sub>2</sub> O) <sub>0.070</sub> ] <sub>x</sub>	1.2 x 10 <sup>3</sup>
<u>cis</u> -[CH(HClO <sub>4</sub> ) <sub>0.127</sub> (H <sub>2</sub> O) <sub>0.297</sub> ] <sub>x</sub>	1.2 x 10 <sup>3</sup>

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TABLE I (continued)

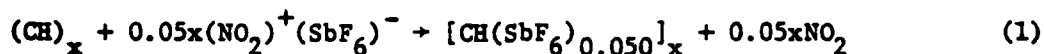
B. n-Type (electron-donating) Dopants<sup>c</sup>

<u>cis</u> -[Li <sub>0.30</sub> (CH)] <sub>x</sub>	2.0 x 10 <sup>2</sup>
<u>cis</u> -[Na <sub>0.21</sub> (CH)] <sub>x</sub>	2.5 x 10 <sup>1</sup>
<u>cis</u> -[K <sub>0.16</sub> (CH)] <sub>x</sub>	5.0 x 10 <sup>1</sup>
<u>trans</u> -[Na <sub>0.28</sub> (CH)] <sub>x</sub>	8.0 x 10 <sup>1</sup>

- a) "cis" or "trans" refers to the principal isomeric composition before doping  
 b) composition by elemental analysis except where stated otherwise  
 c) composition by weight uptake  
 d) dopant used: (SO<sub>3</sub>F)<sub>2</sub>. No composition or analysis given. Anderson, L.R., Pez, G.P., and Hsu, S.L.: 1978, J.C.S. Chem. Comm., pp.1066.  
 e) by electrochemical doping using [(n-C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>N]<sup>+</sup>[PF<sub>6</sub>]<sup>-</sup>. Nigrey, P.J., MacDiarmid, A.G., and Heeger, A.J.: 1979, unpublished observations.

Dopant pressures  $\leq 1$  torr are usually satisfactory. With many dopants the conductivity increases rapidly through the semiconducting regime to the metallic regime. The concentrations of the dopants given in Table I are generally the maximum or close to the maximum value readily obtainable. Doping can be terminated at any degree of lower doping level desired, with corresponding lower conductivity.

Salts containing the (NO)<sup>+</sup> or (NO<sub>2</sub>)<sup>+</sup> ions also act as good dopants (10). For example, the SbF<sub>6</sub><sup>-</sup> group can be introduced readily into (CH)<sub>x</sub> simply by treating a (CH)<sub>x</sub> film (ca. 85% cis isomer) with a CH<sub>3</sub>NO<sub>2</sub>-CH<sub>2</sub>Cl<sub>2</sub> solution of the appropriate salt. Thus, (NO<sub>2</sub>)<sup>+</sup>(SbF<sub>6</sub>)<sup>-</sup> yields golden, flexible, highly conducting films of [CH(SbF<sub>6</sub>)<sub>0.05</sub>]<sub>x</sub>, (Table I) with liberation of NO<sub>2</sub>, viz.,



It has been found very recently that (CH)<sub>x</sub> films may be doped electrochemically either to the semiconducting or metallic regime (13). This is a most important development since it opens up a general, very simple, readily controllable means of doping with a wide variety of species which can not be introduced by an obvious conventional chemical means. For example, it was found that when a strip of (CH)<sub>x</sub> film (ca. 82% cis-isomer) was used as the anode in the electrolysis of aqueous 0.5M KI solution with a potential of 9 V. it was doped during ca. 0.5 hour to the metallic state, to give, by elemental analysis, (CHI<sub>0.07</sub>)<sub>x</sub>. It is important to note that the flexible, golden-silvery films contained no oxygen (total C, H, and I content=99.8%) and hence had undergone no hydrolysis and/or oxidation during the electrolytic doping process. When the (CH)<sub>x</sub> was used as the anode in the electrolysis of 0.5M [(n-C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>N]<sup>+</sup>[ClO<sub>4</sub>]<sup>-</sup> in CH<sub>2</sub>Cl<sub>2</sub> at 9V., doping occurred during ca. 1 hour to give highly conducting (Table I), flexible films which, by elemental analysis, had the composition [CH(ClO<sub>4</sub>)<sub>0.0645</sub>]<sub>x</sub> (13). Lower doping levels obtained during shorter electrolysis times gave material having conductivities in the semiconductor region. Similar results were obtained by the electrolysis of methylene chloride solutions of [(n-C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>N]<sup>+</sup>[SO<sub>3</sub>CF<sub>3</sub>]<sup>-</sup> and

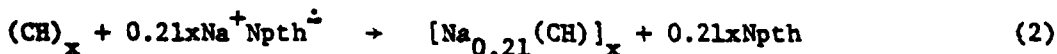
$[(n-C_3H_7)_3NH]^+[AsF_6]^-$  both of which gave highly conducting golden-silvery flexible films. The former is assumed to contain the  $(SO_3CF_3)$  and the latter, the  $(AsF_4)$  species, since elemental analysis of the film gave a composition corresponding to  $[CH(AsF_4)_{0.077}]_x$ . The  $(AsF_4)$  is probably formed by a reaction sequence involving proton abstraction from  $[(n-C_3H_7)_3NH]^+$  by fluorine atoms from  $AsF_6$  during the electrolysis process (13).

(ii) Nature of the  $(CH)_x$  Chains and Dopant Species. Raman studies show that the iodinated and brominated films should be formulated as  $[(CH)^+(X_3)^-]_x$  where  $X=Br$  or  $I$ , at least a significant portion of the halogen being present as the  $X_3^-$  ion (14). The halogen partly depopulates the  $\pi$  bonding system and oxidizes the  $(CH)_x$  to a polycarbonium ion chain. This conclusion is supported by carbon 1s core shifts from ESCA studies (15). The  $(NO)^+$  ions are also excellent species for oxidizing the  $\pi$  system of  $(CH)_x$  and are capable of concomitantly introducing anions which stabilize the polycarbonium ion chains (10). For example, the  $[CH(SbF_6)_{0.050}]_x$  species given in equation 1 is more appropriately formulated as  $[CH^{+0.050}(SbF_6)_{0.050}]_x$ .

The most simple and general method for simultaneously oxidizing the  $(CH)_x$   $\pi$  system and introducing stabilizing anions appears to be that involving electrochemical doping (13). Thus, species such as  $[CH(ClO_4)_{0.0645}]_x$ ,  $[CH(AsF_4)_{0.077}]_x$ , etc. formed electrochemically as described above are believed to contain the  $(ClO_4)^-$  and  $(AsF_4)^-$  ions, respectively, although the extent to which charge transfer to the anionic species occurs may be expected to vary according to the nature of the dopant.

#### (B) N-Type Doping.

Electron-donating, i.e. "n-type" dopants, may also be introduced into  $(CH)_x$  films (16) (Table I) simply by immersing the film in a THF solution of e.g. sodium naphthalide, viz.,



A very large increase in conductivity is noted but it is not as great as that observed with most p-type dopants. Alkali metals may also be introduced by, for example, allowing a liquid sodium/potassium alloy at room temperature, or molten potassium to contact a  $(CH)_x$  film (17). A liquid sodium amalgam will also Na-dope the film at room temperature (17). Preliminary experiments indicate that the  $(CH)_x$   $\pi$  system may also be reduced electrochemically to give n-type doping by, for example, the electrolysis of a solution of LiI in THF using a  $(CH)_x$  film as the cathode, to give  $[Li^+(CH)^-]_x$  films (13). The  $(CH)_x$  chain in these materials may be considered as a polycarbanion associated with the corresponding  $M^+$  metal ion.

## 2. PHOTOELECTROCHEMICAL REACTIONS AT POLYACETYLENE INTERFACES.

A chemical reaction involving a reduction process, e.g.,





can take place with the concomitant production of an electric current when an undoped (p-type)  $(CH)_x$  film, immersed in a solution containing the oxidized and reduced forms of an appropriate couple, is irradiated with light of appropriate wavelength (18). In the case of the polysulfide system, the reverse (oxidation) process,



will take place simultaneously at the counter electrode, e.g., Pt, which is not irradiated. The ions produced at a given electrode then diffuse to the other electrode and become available for reuse at that electrode as shown in Figure 1. The process is, therefore, continuous as long as the  $(CH)_x$  electrode is irradiated with light of appropriate wavelength (18,19). A photovoltaic effect can be observed ( $V_{oc} \sim 0.3$  volts under illumination of ca. 1 sun) even with the simple set-up shown in Figure 1 if a fairly thick film of trans- $(CH)_x$  is used in order to reduce somewhat the otherwise high resistance of the  $(CH)_x$  electrode. By using a different cell configuration, described in detail elsewhere (18,19), an open circuit current of ca. 40  $\mu$  amps/cm<sup>2</sup> may be obtained. This will undoubtedly be increased by using partially doped  $(CH)_x$  and thinner films. Since  $(CH)_x$  is a p-type semiconductor, photo-generated electron-hole pairs become separated at the  $(CH)_x$ -electrolyte interface and electrons are injected into the electrolyte as shown in Figure 2. It seems highly likely that it should be possible to fabricate a variety of photoelectrochemical photovoltaic cells using  $(CH)_x$  electrodes immersed in aqueous or nonaqueous solutions of appropriate redox couples.

### 3. CONCLUSIONS

It can be seen clearly that  $(CH)_x$  is quite remarkable in that its conductivity can be readily modified to span an extraordinarily large range. Considering possible polyacetylene derivatives, replacement of some or all of the hydrogen atoms in  $(CH)_x$  with organic or inorganic groups, copolymerization of acetylene with other acetylenes or olefins, and the use of different dopants should lead to the development of a large new class of conducting organic polymers with electrical properties that can be controlled over the full range from insulator to semiconductor to metal. Furthermore, there is considerable potential for the possible application of parent or doped  $(CH)_x$  to the fabrication of various types of electronic devices, solar cells, etc.

### 4. ACKNOWLEDGMENT

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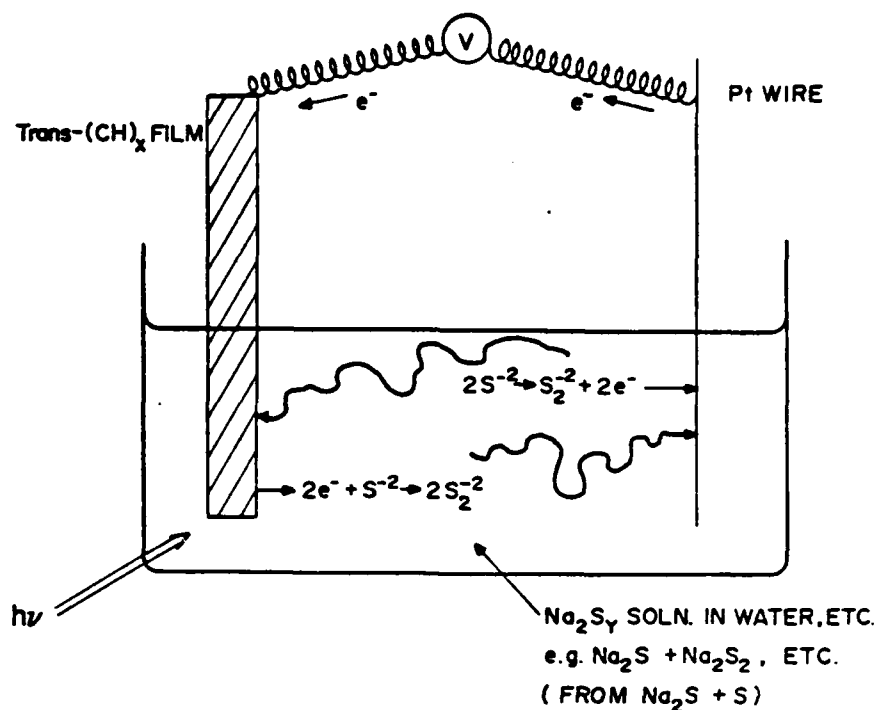


Figure 1. Simple (CH)<sub>x</sub>/sodium polysulfide photoelectrochemical photovoltaic cell.

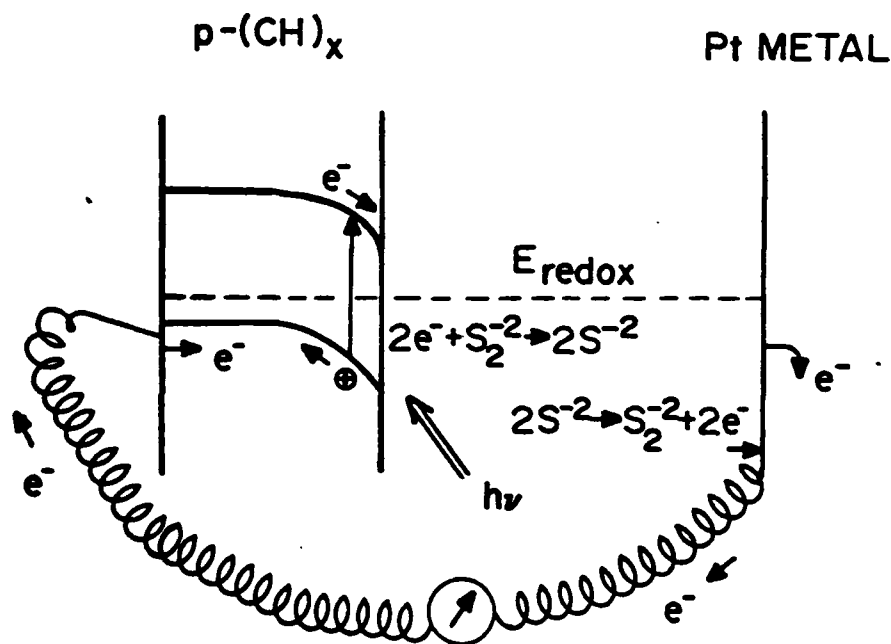


Figure 2. Electrode processes in a (CH)<sub>x</sub>/sodium polysulfide photoelectrochemical photovoltaic cell.

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